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RESEARCH AND DEVELOPMENT REPORT

TEXTILE SERIES - REPORT NO. 80

THE TENSILE BEHAVIOR
OF SOME PROTEIN FIBERS

by
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and
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DEPARTMENT OF THE ARMY
OFFICE OF THE QUARTERMASTER GENERAL

Office of the Quartermaster General
Research and Development Division
Textiles, Clothing, and Footwear Branch

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Foreword

Because of its wide use in U. S. Army clothing, wool is a strategic material. It is one of the oldest textile fibers, yet there is still much to learn about its properties. What makes wool such a desirable fiber and how and to what extent it may be replaced in the event of emergency wool shortages is the subject of much Quartermaster textile research.

In this study, testing methods developed during the past years at the Quartermaster Research and Development Laboratories, Philadelphia, Pa., have been used to characterize wool along with some other natural and man-made protein fibers chemically similar to wool: mohair, human hair, casein, Vicara, and silk. The tensile properties of these fibers, including their recovery, have been investigated in the original state, after repeated stresses (mechanical conditioning), and when swollen in water. It was expected that the reasons for the outstanding performance of wool in service would come to light in tests under such varied conditions.

The results of this investigation clearly show the excellent recovery of wool even after high stresses and strains and its enhanced recovery when wet. They also demonstrate the mobility of wool single fibers in the yarn structure. These characteristics make wool unique among the textile fibers; this work shows that the properties of wool in these respects are not duplicated even in other protein fibers.

Two other Textile Series Reports have discussed the tensile characteristics of a number of textile fibers including protein fibers. "Tensile Recovery Behavior of Textile Fibers" (No. 64) presented data on the recovery characteristics of the fibers in their original state, and "Mechanical Conditioning of Textile Fibers" (No. 82) showed the effect of repeated stresses on tensile behavior. A future report will discuss in more detail the effect of water on fiber properties. The purpose of the present report is to give a comprehensive view of the tensile properties of wool and other protein fibers when tested under all these conditions.

We are indebted to the following organizations for supplying the samples tested in this program: Belding-Heminway Co., Inc., New York 18, N. Y.; Eastern Regional Research Laboratory, U. S. Department of Agriculture, Philadelphia, Pa.; Forstmann Woolen Co., Passaic, N. J.; Goodall-Sanford, Inc., Sanford, Me.; Virginia-Carolina Chemical Co., Taftville, Conn.; Francis Willey and Co., Boston, Mass. The permission of Mr. H. Hindman, Instron Corp., Quincy, Mass., to use a special attachment to the Instron tester

for the performance of wet yarn tests is appreciated. Thanks are due to Miss R. L. Freedman for her cooperation in the experimental work, to Mr. J. Medernach for the preparation of the figures, and to Mr. N. E. Roberts for reviewing the paper. We also wish to thank Dr. W. J. Hamburger, Mr. G. E. Hopkins, and Mr. A. M. Sookne for their helpful criticisms on the interpretation and presentation of the data.

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Abstract

The tensile properties of wool, mohair, human hair, casein, Vicara, and silk including their behavior when knotted, after repeated stresses (mechanical conditioning) and when swollen in water were investigated. The immediate elastic recovery, delayed recovery and permanent set of these fibers were measured by a cycling technique using the Instron tensile tester and the Sookne-Harris fiber extensometer. Actual and relative values of elongation components under different testing conditions are demonstrated graphically from the beginning of the stretching procedure up to rupture.

Wool has an outstanding elastic recovery in the dry state and its elastic behavior is improved after mechanical conditioning and when wet. Mohair, human hair, and to some extent casein and Vicara, have elastic properties similar to wool. The tensile behavior of silk is different, being characterized by considerable permanent set (dry and wet) and by high immediate elastic recovery after mechanical conditioning.

I INTRODUCTION

Wool is the most important protein fiber,* and has a combination of extraordinary properties not found in any other natural, man-made, or synthetic textile material. This study is confined to the tensile properties - i.e., tenacity, extensibility, and elastic recovery - of wool and other protein fibers. Major emphasis is placed on recovery, since the high resilience of wool is to a large extent responsible for its outstanding performance. [12, 30] The behavior of wool will be compared to that of five other natural and regenerated protein fibers: mohair, human hair, casein, Vicara and silk. These fibers are similar to wool, in that they consist of long-chain protein molecules, although some differences exist in their chemical composition, crystallinity and crystal structure, and they are also considerably different in morphology. The aim of this study is to learn to what extent wool-like tensile properties are present in these closely related fibers.

II TESTING EQUIPMENT

Tensile tests were made using two instruments: the Instron tensile tester, Model TT-B and the Sockne-Harris fiber tester. Both were used to determine stress-strain characteristics including recovery behavior, by differentiating between three elongation components.

A. Instron Tensile Tester

The Instron tensile tester [5, 11] is a versatile instrument having a high extending range (maximum 40 in. less the length of the jaws), a large loading capacity (from 2 g up to 454 kg) and wide jaw speed variability (from 0.02 in. to 20 in. per minute in twelve steps). Load is measured by an electric strain gage cell which activates a recorder pen to reproduce the stress-strain relationship continuously. The tester operates at a constant rate of elongation. A relatively large initial gage length (5 in.) and a high rate of jaw separation were used. The latter was limited by the inertia of the extending and recording mechanism. This tester served for most of the investigations reported in this paper, since it is a convenient and accurate testing instrument for single fibers as well as for yarns. Knot behavior and the effect of repeated stresses were investigated exclusively with the Instron tester.

* Protein fibers are built up from amino acids and they contain mainly carbon, hydrogen, oxygen and nitrogen. Fiber-forming proteins consist of long-chain molecules held together by peptide linkages, i.e., by the condensation of amino and carboxyl groups. Neighboring long-chain molecules are also cross-linked in protein fibers.

B. Sookne-Harris Fiber Tester

The Sookne-Harris tester [25, 26, 34] is a balance designed for testing single fibers. One arm holds the fiber; to the other arm a variable weight is attached in the form of a chain. This chain hangs in a catenary curve, one end fixed on the balance arm, the other moving vertically to maintain equilibrium in the balance. Variation in load capacity is obtained by calibrated chains of different weights. The fiber is mounted between the end of one balance arm and a vertically moving platform by two anvil type jaws. As elongation of the fiber creates a tension on one end of the balance, equilibrium is restored automatically by a photo-electric cell through chain length adjustment. A pen fixed to the moving end of the chain records stress-strain curves continuously on a drum revolving synchronously with the extending mechanism. The relatively short distance between the end of the balance arm and the platform (approximately 4 in.) limits the initial gage length (usually 2 in.). The tester operates at a constant rate of jaw movement which is reversible. The highest rate of elongation was used which is much less than that used on the Instron tensile tester. The Sookne-Harris tester served exclusively for the testing of single fibers; it was especially valuable for the testing of such fibers immersed in water.

It would not be surprising if results obtained by such different instruments would deviate somewhat from each other, reflecting the influences of the tester and of the particular conditions of the tests. These influences were, however, relatively slight for the tests described in this paper and results obtained by the two instruments are comparable.

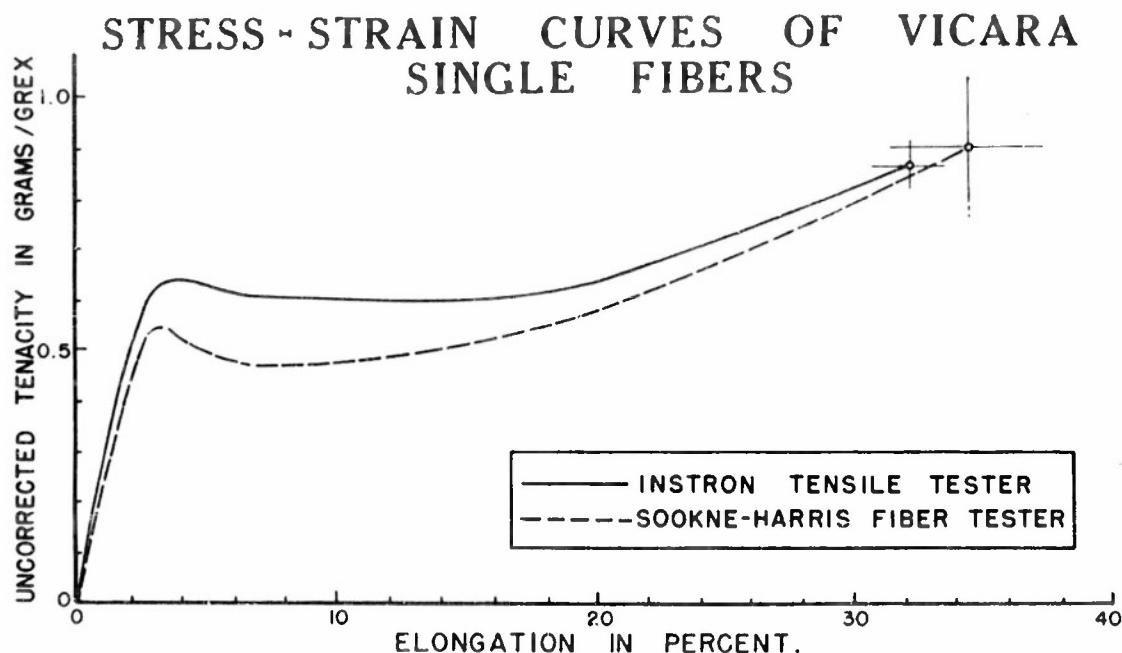
III TESTING TECHNIQUE AND PRESENTATION OF DATA

A. Stress-Strain Relationship

Stress-strain properties in the original state were tested under standard conditions at 70 F and 65 percent relative humidity. A 5-in. gage length and a jaw separation rate of 5 in. per minute, corresponding to 100 percent elongation of the specimen per minute, were used for the Instron tensile tester. Since this could not be duplicated in the Sookne-Harris tester, the longest practical gage length, usually 2.0 in., and the highest jaw speed were used, corresponding to a 10 percent elongation of the specimen per minute.

First, load-elongation curves were made from 10 or 20 individual specimens, depending on the uniformity of the material, from which the mean values of load and elongation at break were computed. A curve with ultimate values closest to the mean was

FIG. 1



then selected from the load-elongation curves obtained, to represent the average stress-strain curve of the material. From this a new stress-strain curve was drawn in which tenacity values were based on the initial fineness expressed in grex according to the recommended practice of the American Society for Testing Materials, [2, 23] and elongation was expressed as percent increase of the original length. As a measure of the uniformity of the samples and of the reproducibility of tests, the standard deviations of tenacity and elongation at break were computed, and these were represented by intersecting vertical and horizontal lines at the breaking point of each stress-strain curve.

In Figure 1 are shown such stress-strain curves for Vicara as obtained on the Instron and the Sookne-Harris testers. It can be seen that the curves obtained by both instruments are essentially identical.

Stress-strain properties under standard conditions reveal to only a limited extent the tensile behavior of fibers unless the tests are repeated under varied humidities and temperatures, after knotting, after repeated stresses and after wetting. Although investigations could not be carried out under all these conditions, tests were made on knotted fibers, on repeatedly extended fibers, and on wet fibers.

B. Knot Behavior

Knot tests have been included in this study, since poor knot behavior is an indication of some inherent weakness of the fiber not observable on the conventional stress-strain curve. It is known that knot tests of some materials, such as viscose, correspond fairly well to their behavior in practical use. [4]* Knot tests also supply information on transverse properties, because they reflect the response of fibers not only to tension, but to bending, compression, shear, and to possible high stress concentration.

The knots were overhand knots tightened mechanically with the Instron tensile tester to improve the reproducibility (which is otherwise rather low). An 8-in. sample, loosely knotted, was first subjected to three consecutive loading cycles at a low jaw speed (0.5 in. or 6.3 percent elongation per minute) to a stress of 5 or 10 percent of the average breaking load, depending on fiber stiffness. This tension was sufficient to obtain a tight knot but did not exceed the elastic limit. The knotted fibers were then cut out from the jaws and relaxed without tension for one hour. They were then tested under the conditions described above in the Instron tester. The very small increase in actual gage length due to the knot was neglected.

C. Mechanical Conditioning

Textile fibers in their practical use are almost without exception subjected to repeated stresses and strains. When they exceed the elastic limit of the fibers, these stresses and strains substantially change the tensile properties. The result of such mechanical conditioning is the elimination of permanent set evidenced by a considerably lower extensibility. Sometimes the tenacity is also affected, being either increased as a result of stress hardening or decreased as a result of weakening or fatigue. The initial modulus is usually higher if stress hardening occurs and lower if the fiber is weakened. It is necessary to investigate the extent of these changes since textile materials react differently after they have been extended. [28]

The samples were mechanically conditioned by extending them 50 times in the Instron tensile tester, from zero tension to an elongation corresponding to 80 percent of the elongation at break, using an initial gage length of 8 in. and a jaw movement of 5 in. per minute. After this operation the samples were cut out from the jaws and allowed to relax for one hour without tension to provide for recovery and to permit an

* Loc. cit., page 267

equilibrium to be attained. The increased fiber length was measured before and after this relaxation period. Then the samples were tested in the Instron tensile tester under the previously described test conditions.

D. Wet Behavior

The tensile properties of protein fibers are easily affected by changes in humidity. For a thorough knowledge of tensile properties, therefore, tests should be conducted at humidities different from the standard 65 percent relative humidity and in the wet state. Although tests were not made at varied humidities, they were made on wet fibers, which represent an extreme in moisture uptake. These tests indicate the direction of change in fiber properties at increased humidities. Wet tests of yarns were carried out on the Instron tensile tester using the standard gage length and speed. Samples of approximately 10 in. in length were immersed in water containing a small amount of detergent (Duponol) for an hour previous to testing. (This procedure caused some of the yarns, unbalanced in the wet state, to unravel since no tension was applied.) The wet samples were then tested in the Instron tensile tester, being protected from drying by a wet fabric cover entirely surrounding the specimen during the tests. Tenacity values of these tests were based on the original fineness in the dry state. Elongation, however, was correlated to the initial wet length which was not significantly different from the dry length.

Water-swollen single fibers dried out too quickly to permit testing by this technique. Single fibers of wool, mohair, human hair, and Vicara were therefore tested under water using the Sookne-Harris tester. The fiber was fixed in the jaws dry (gage length 1.5 in.), immersed in water, and tested after one minute immersion (with a jaw speed of 0.2 in. per minute corresponding to 13 percent elongation per minute). Tenacity and elongation values of wet single fibers were based on the initial dry fineness and dry length respectively.

E. Tensile Recovery

An initial study of tensile behavior must include recovery, since it is important to know to what extent an imposed strain may recover when the tension is released. Recovery tests of textiles have been made mostly by separating the recoverable and the non-recoverable elongation or permanent set. Differentiation between two elongation components, however, is not sufficient to characterize the complex response of visco-elastic materials to an imposed stress or strain. It is also necessary to take the time factor into consideration, and to differentiate between that part of the elongation which recovers immediately and that which recovers after a longer period.

Recovery tests of fibers in the original state, after mechanical conditioning, and when wet, were performed using a repeated cycling technique previously described [29] to separate three components of the total elongation: immediate elastic recovery, delayed recovery and permanent set. Using the Instron tester, recovery tests were made on 10 to 15 individual samples, each extended to a progressively increased strain. The above described gage length and jaw speed, immediate removal of elongation after extension, and a recovery time of five minutes were used. Under these conditions, immediate elastic recovery is that part of the total elongation which takes place in the range of a fraction of a second to a few seconds after the release of strain. Delayed recovery, or primary creep, is the additional recovery which is attained after a relaxation period of five minutes. Permanent set is that part of the total elongation which does not recover in five minutes and can be considered, in the first approximation, as permanent. Some authors designate this component as "semi-permanent set" and others as "temporary set" or "secondary creep."

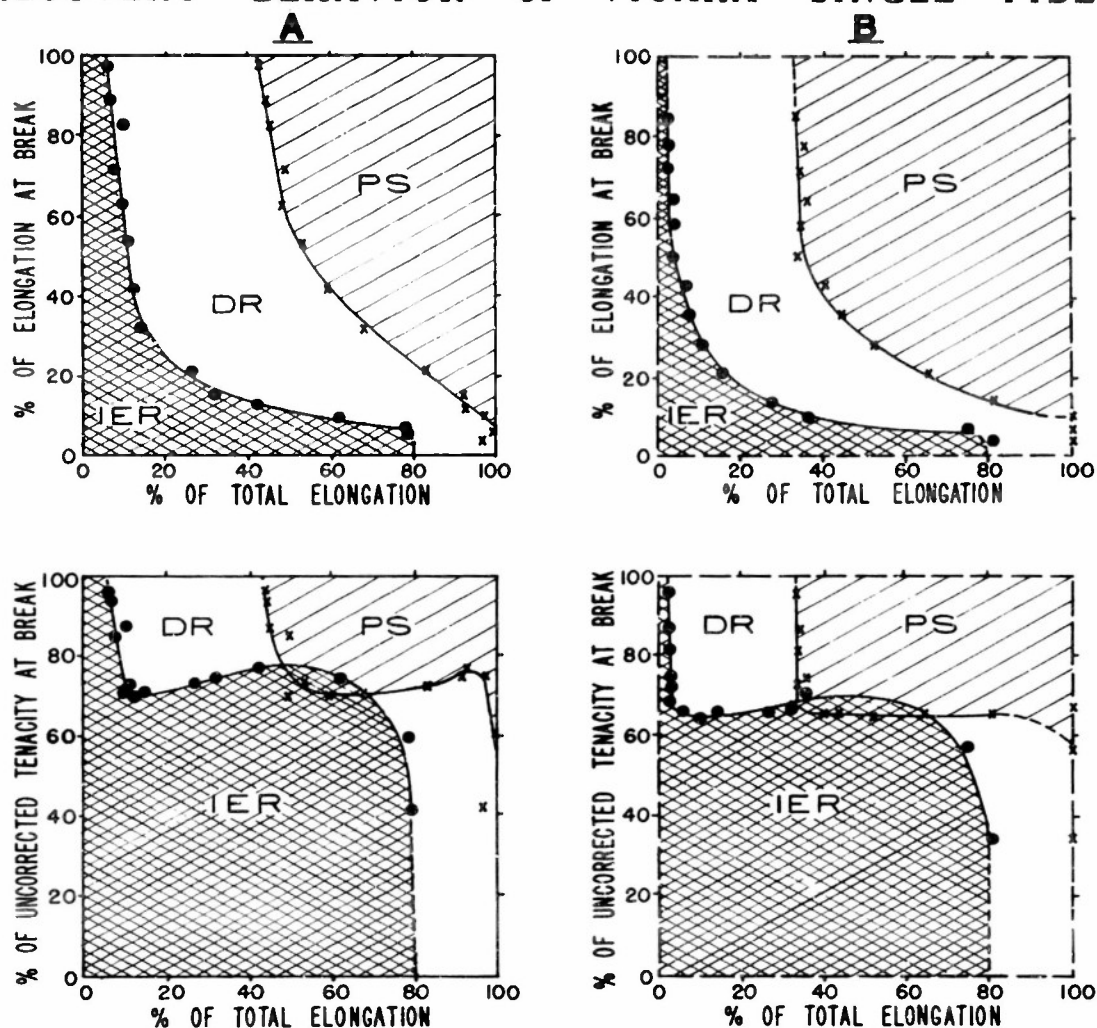
The Instron tensile tester was used for all recovery tests except for those of wet single fibers. Recovery tests of mechanically conditioned samples were made after they had been previously extended 50 times to 80 percent of their elongation at break and relaxed for one hour without tension. Recovery of wet yarns was tested after the yarns had been water-soaked for one hour. They were protected from drying out during the test by a wet fabric cover.

The recovery of wet single fibers was tested under slightly different conditions in the Sookne-Harris tester. Only one specimen was repeatedly extended to increasing strain values up to the breaking point. The specimen was inserted in the tester dry and then immersed in water. It remained under water during the test which was performed one minute after immersion. The following test conditions were observed: gage length of 1.5 in., platform travel of 0.2 in. per minute, immediate removal of strain after extension, recovery time of one minute.

The recovery of fibers can be expressed at different stress or strain values either by actual values (percent of the three elongation components correlated to the initial length) or by relative values (percent of each elongation component correlated to the corresponding total elongation). The entire recovery behavior of a fiber is shown best by graphs. A simple demonstration of tensile recovery is achieved by representing the actual values of the three strain components at different stress and strain levels by three separate curves. The superimposition of these three curves gives then the conventional stress-strain curve. Since this demonstration has some disadvantages, [29] two other types of graphs, rectangular and quadratic, have also been used in this study to

demonstrate the recovery behavior of the fibers tested. In both, not actual but relative values of strain components are shown and the three elongation components are represented as areas. Recovery data are demonstrated by four curves in Figures 3 and 7, by rectangles in Figure 8, and by quadrats in Figures 2 and 9.

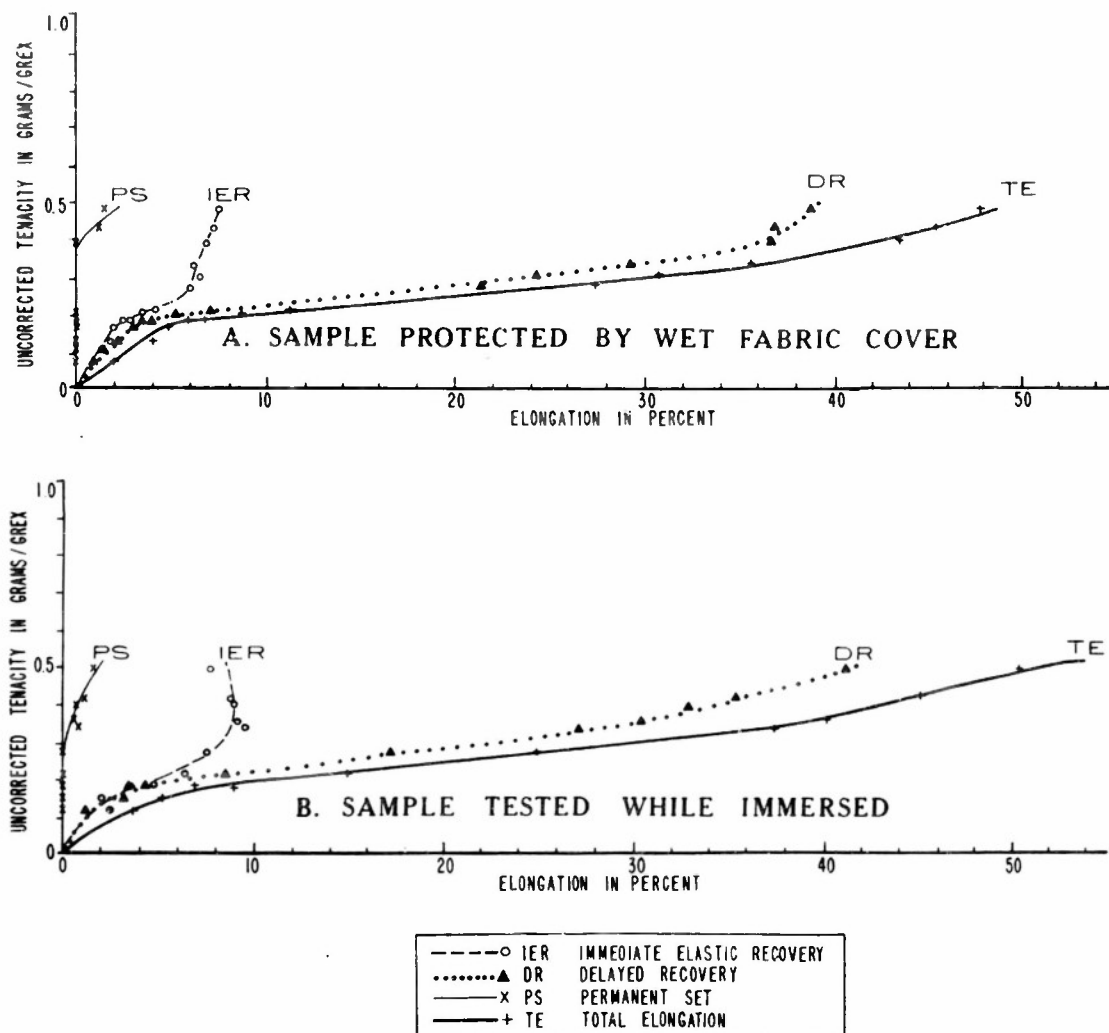
FIG. 2
RECOVERY BEHAVIOR OF VICARA SINGLE FIBERS



A. TESTED WITH THE INSTRON TENSILE TESTER.
B. TESTED WITH THE SOOKNE-HARRIS TESTER.

▨ PERMANENT SET □ DELAYED RECOVERY ▩ IMMEDIATE ELASTIC RECOVERY

FIG. 3
RECOVERY BEHAVIOR OF WET WOOL YARN



The comparability of the results obtained by the different testing techniques is demonstrated by Figures 2 and 3.

The Instron tensile tester is more suitable for recovery tests than the Sookne-Harris tester since a larger gage length and a higher speed of crosshead travel can be used. The approximation of the true values for immediate recovery depends greatly upon a sufficiently high jaw speed in the recovery cycle. It is also obvious that data obtained on a set of samples, as practiced on the Instron, are more representative since they correspond to the average behavior. The recovery of Vicara single fibers as obtained on the Instron and Sookne-Harris testers is compared by quadrats in

Figure 2. Despite the discussed differences essentially the same values were obtained for delayed recovery by both testers. Lower immediate elastic recovery and higher permanent set were observed, however, when the Sookne-Harris tester was used. This could be expected from the lower jaw speed used for this instrument. When fibers are extended at a lower rate, a higher portion of elongation remains unrecoverable as a result of increased "plastic flow" and slippage.

To remove any question as to the validity of recovery tests using the wet fabric cover, the recovery tests on wet wool yarn were repeated using an attachment to the Instron tensile tester which permits the specimens to be tested under water.* As shown in Figure 3A and B, essentially identical recovery curves were obtained by each method. Minor differences might be caused by the fact that the temperature during the tests on immersed specimens could not be controlled.

IV KNOWN TENSILE PROPERTIES OF PROTEIN FIBERS

An extremely high number of tensile tests of natural and regenerated protein fibers have been conducted, furnishing data on the tenacity and total elongation at break, the stress-strain relationship, the yield point, the initial modulus of elasticity (Young's modulus), the stiffness, and the work necessary for rupture. These have been obtained on a wide range of samples under different conditions and using different types of testing machines. According to these tests wool, mohair, human hair and some of the regenerated protein fibers have high extensibilities (30-50 percent), relatively low tenacities (0.5-1.5 g per grex), [24]** and low initial moduli (24-44 g per grex). [36] As compared to other fibers, they need considerable work to rupture (0.38-0.40 g-cm per grex for a fiber length of one cm). [20] In contrast, natural silk exhibits a lower extensibility (13-20 percent), a higher tenacity (2.5-4.5 g per grex), [24] and a higher modulus (84-120 g per grex). [36] The energy required to rupture silk is also much higher (0.19 g-cm per grex for a fiber length of one cm). [20]

According to data published by Boehringer and Schieber, [4] wool and silk fibers exhibit a relative knot strength of 85 and 88 percent respectively. The corresponding values for knotted regenerated protein fibers vary, being 33 percent for fish albumen fiber, 92 percent for Tiolan and 95 percent for Lanital.

* These tests were made at the Instron Corporation, Quincy, Mass., where the attachment was available.

** Tenacities from 1.12 to 1.21 g per grex for 50's to 80's wool tops and from 1.08 to 1.81 g per grex for a large variety of hairs can be calculated from tensile strength data given in the American Wool Handbook. [31]

Practically no information exists on the tenacity and extensibility of protein fibers after mechanical conditioning.

Sufficient data on the tensile properties of different protein fibers at varied humidities and when wet are available. They generally show a markedly increased extensibility, but decreased tenacity and stiffness at higher humidities or when wet. The influence of swelling in water is more pronounced for regenerated fibers than for native proteins, the relative wet strength of wool being 82 percent,* and of silk 79 percent, but of Tiolan only 35 percent, of Lanital 47 percent, and of fish albumen fiber 59 percent. [4]

In most recovery tests of protein fibers, differentiation has been made only between total recovery and permanent set. Meredith [19] described the tensile recovery of single fibers of 36's, 56's and 64's wool, of mohair, camel hair, silk, and casein, using a repeated cycling technique. He compared their "elastic recovery" (ratio between recoverable and total elongation) to that of other textile fibers at different stress and strain levels. The high recovery of wool and hair, apparent from these tests, was exceeded only by nylon and was not equaled by casein or silk. Maillard and co-workers [17, 18] included single fibers of wool, mohair, and silk in their recovery tests. They observed markedly lower recovery for these fibers than for nylon when recovery is compared on the basis of the ratio between recoverable energy and total energy. Meredith and Peirce [21] investigated the "plasticity" of some protein fibers by "cumulative extension," i.e. by extending fibers repeatedly up to 4 percent elongation and then measuring the total unrecoverable elongation. An increase in permanent set (based on energy imposed) indicated high plasticity of the yarns tested and a failure in recovery from oft-repeated small strains. The lowest plasticity (although not as low as that of linen and nylon) was found for silk, followed by wool, mohair and camel hair; casein showed considerable plasticity like ordinary viscose or acetate. The "plasticity" of wool and human hair has been investigated also by Ripa and Speakman [22] who extended wet single fibers under a constant load (approximately 0.5 g per grex) and held them under tension for a long period of time. The rate of length increase was considered as a measure of plasticity and was found to be governed by the sulfur or cystine content of the fiber. High plasticity was observed at low sulfur contents and interpreted as breakdown of disulfide bonds. Plasticity is, however, not determined solely by the sulfur content of keratin fibers, but it is also influenced by other factors such as high ratio between amorphous and crystalline portions of the fiber.

* "Relative wet strength between 78 and 85 percent are listed for 36's to 80's standard wool tops in the American Wool Handbook. [31]

Data on total recovery of wool and regenerated protein fibers when wet were published by Speakman [27] as early as 1926, recently by Maillard and co-workers, [18] Harris and Brown, [9] and Sookne, [26] the latter three authors using the Sookne-Harris tester. It became apparent from these tests that the recovery of wet wool fibers is exceptionally high, well exceeding that of the dry fibers. This high recovery was shown by a cycling technique in which nearly complete recovery on the unloading curve was apparent even when extended close to the breaking point. The recovery behavior of wet wool is, therefore, in contrast to that of many other textile materials. Some fibers (e.g., acetate) show an increased extensibility when wet and this is attained by a higher portion of nonrecoverable elongation. The recovery of wet regenerated protein fibers is similar to wool, though markedly lower in value. [9]

Recovery tests at 60 and 90 percent relative humidities for elongations between 1 and 10 percent published recently by Beste and Hoffman [3] are noteworthy since they indicate considerable increase of recovery for wool, a slight increase for silk and a decrease for casein, viscose, and acetate at the higher humidity.

Hamburger, Morgan and Platt [8] investigated the immediate elastic deformation of wool, mohair, and silk single fibers at elongations up to 5 percent using the sonic modulus technique. [7] The highest actual values at 3 percent elongation were observed for mohair, followed by wool and silk, these exceeding even nylon and Dacron. At the elongation of 5 percent Dacron had the highest immediate elastic deformation, followed by mohair, silk, wool and nylon.

Data differentiating between immediate recovery, delayed recovery and permanent set at any stress and strain value up to the breaking point are known from cycling tests for some protein fibers: worsted wool yarn, silk, raw and stabilized casein multifilament. [29] Their behavior was compared to that of a large number of other textile fibers, showing that only nylon is similar to wool in recovery. In both materials delayed recovery markedly exceeds, however, the immediate elastic recovery (at least from the yield point up to the breaking point).

Information on the three strain components of protein fibers after repeated stresses or when wet is nonexistent, but is necessary for an overall picture of the tensile properties of these textile materials.

V MATERIALS TESTED AND THEIR STRUCTURAL PROPERTIES

Eight protein fibers were tested in the form of single fibers, multifilaments, or staple yarns. Behavior in the two latter forms reflects inherent fiber properties only partly, because of the effect of the yarn structure.

Wool is the most important protein fiber and was tested as a single fiber (50's) and as a worsted staple yarn (28.4/1-R25 containing 64's). It is obvious that two samples cannot represent the large variety of wool fibers which may differ in origin, grade, staple length, etc., or of wool yarns which may differ as well in count, twist, construction, processing, finishing, etc. Nor can they be considered as average wool samples, since a single material cannot represent all possible variations. The samples tested however, are not uncommon, and although they admittedly show the properties of only one wool staple fiber and one worsted yarn, they are not essentially different from the bulk of other wool samples.

Wool consists of long-chain molecules of the water insoluble polypeptide keratin, which is also the main constituent of feathers, horn, and epidermis. It is chemically a very complicated substance, built up by condensation of about twenty different amino acids. [6] Fifty percent of these amino acids consist, however, of glutamic acid, cystine, leucine and arginine. Many of the amino acid residues have bulky side chains and they have not only amino groups but reactive hydroxyl, carboxyl, amide, and disulfide groups which are apt to form cross links between neighboring polypeptide chains. It is known that sufficiently strong cross links prevent slippage of long-chain molecules in the stretching procedure and that they also facilitate the return of the molecular arrangement to the original structure when the extending force is released. The disulfide cross linkages of cystine are considered to be mainly responsible for the recovery of wool besides linkages between polar groups (OH, NH₂, and COOH) and hydrogen bonds. The high recovery of wool is also due to the flexibility of the keratin molecules themselves. When stretched, the molecular structure transforms from the folded alpha form (with the fiber period 5.1 Å, 3 x 1.7 Å) to the unfolded beta form (with the fiber period of 6.7 Å, 2 x 3.34 Å). In unstretched wool the alpha keratin crystallites are randomly distributed, and even beta keratin (in highly stretched wet wool) is only moderately oriented when compared to other highly oriented fibers such as ramie, Fortisan, stretched nylon, Dacron polyester fiber, and polyethylene.

The decreased tenacity and the increased extensibility of wet wool is evidence of a weakening caused by the affinity of the wool substance to water. Swelling of wool results in increased recovery, since the polypeptide chains can respond more easily by folding and unfolding to relieve tension. Some cross linkages (hydrogen bonds and bonds between polar groups) are weakened in the swollen state, while other cross linkages (disulfide bonds) remain active. When these stronger bonds are broken by hydrolysis (at higher temperature, in the presence of alkalies and acids, or by reduction), wool shows properties like regenerated protein fibers: lower tenacity, higher extensibility, lower recovery, and increased permanent set. Wool is complex, not only chemically and in its crystal structure, but also morphologically since it is built up from different layers: cuticle, cortex (approximately 90 percent of the wool fiber) and sometimes medulla, besides the recently discussed "epicuticle." Wool fibers are more or less crimped and their surface is not smooth, due to scales upon which the felting of wool principally depends. These latter morphological properties contribute much to the mobility of wool fibers in textile structures.

Mohair is most closely related to wool of all the fibers tested. Its basic substance, keratin, is identical chemically and also in crystallographic structure to wool keratin. The main difference between mohair and wool is in its morphology, mohair being coarse, longer with less crimp, and less pronounced scales. A higher sheen and more difficult dyeability are also characteristic of mohair. This fiber occupies an intermediate position between wool and hair sorts. Mohair was tested in the form of a single fiber (32's, Texas) and a staple yarn (28/1 containing 65 percent 32's and 35% 28's, Texas). Their fineness was similar to the corresponding wool samples tested. The twists of wool and mohair yarns were also similar.

Human hair is essentially the same as wool and mohair in chemical composition except for its higher cystine content which is responsible for its higher stability and lower "plasticity." It is morphologically very different from wool, being coarser, longer, and having a smooth surface and no crimp. The hair tested was fair, and came from a 12 year old girl. It was investigated in its original state without the usual extraction and washing procedure to eliminate the grease in order to avoid any possible damage or changes in its mechanical properties by these operations.

Casein represents the best-known and most easily handled man-made protein fiber. [24] It corresponds to Aralac, to the Italian Lanital, to the German fibers Tiocell, Tiolan, Lactofil, to Cargan from Belgium, and to Casolana from Holland. Casein fibers are made from the phosphor-protein of milk containing approximately 80 percent alpha (soluble) and 20 percent beta (insoluble) substance. The chemical treatment, with even mild alkalies, necessary to obtain spinnable solution causes a

shortening of the original polypeptide chains and breakdown of sulfur cross bonds. This explains the lower strength, poorer recovery, and higher permanent set of wet casein as compared to wet wool. The molecules of casein are straightened out in the spinning procedure and show the X-ray pattern of beta keratin. Casein was investigated in the form of unstabilized multifilaments (300/40).

Vicara is a recently developed fiber containing the rather hydrophobic protein of corn zein. [14, 35] It is obtained by extrusion from a solution followed by stretching, curing with formaldehyde and drying of the filament. It was tested as monofilament.

The properties of casein and Vicara may serve to indicate the tensile behavior of other man-made protein fibers for which the generic term Azlon was proposed by the American Society for Testing Materials. [1] Such fibers are: [24, 37] Ardil and Sarelone from peanuts; Soyline from soybeans; Rislan from castor beans; and fibers from regenerated wool in the alpha or in the beta form, from collagen, fish protein, egg white, edestin, cotton or tobacco seed globulin, and to some extent even from solubilized silk fibroin. [33] Globular proteins are not fiber forming. Egg albumin is originally spherical and can be spun into fibers only after "denaturation." [15] Most man-made protein fibers are weaker than natural fibers, especially when wet, and they lack the high wet recovery of native keratins, even after stabilization with curing agents such as formaldehyde. [16]

Natural silk contains originally two proteins, sericin and fibroin of which only the latter is in degummed silk fibers. Silk is chemically, morphologically, and in its crystallographic structure very different from wool and less complex in many respects. Silk fibroin [10, 13, 39] is built up mainly of four amino acids: glycine, alanine, tyrosine, and serine. They make up 90 percent of the total amino acid residues. They are not very different from each other and have only small side chains; thus most of the hydroxyl, basic and acid groups are accessible. The smooth long-chain molecules are close together and cross-linked by relatively weak hydrogen bonds; no sulfide cross-bridges are present. The rod-like molecules are fully extended, the fiber period being 6.8 Å, 2 x 3.4 Å. A large portion of the fibroin is crystallized and the polypeptide chains are in alignment with the fiber axis. The X-ray diagram of silk shows therefore a high degree of orientation, comparable to that of other highly oriented textile fibers. According to this structure, silk has higher tenacity but lower extensibility and lower recovery than wool and it does not swell as much in water as native or regenerated protein fibers. Silk was tested in the form of a fairly uniform multifilament containing 132 individual fibers.

VI RESULTS

A. Stress-Strain Properties

Although some of the stress-strain properties reported in this study are known, their investigation was a prerequisite for the correlation of their recovery with stress and strain values.

The stress-strain relationship of the fibers in the original state and when knotted is shown in Figure 4, after repeated stresses in Figure 5, and when wet in Figure 6. Table I contains the characteristics of the samples tested and numerical data of the stress-strain curves represented in Figures 4, 5, and 6.

The following additional data are listed in Table I:

Density. The density may be used to transform the tenacity values to tensile strength values by the following relations: Tenacity (in g per grex) x density x 10 = tensile strength in kg per sq mm. Tenacity (in g per grex) x density x 14223 = tensile strength in lb per sq in.

Corrected breaking tenacity. These values are based on the diminished actual fineness at the breaking point and are obtained by the relation

$$T_C = T_U \times \frac{100 \times E}{100}$$

where T_C = corrected tenacity in g per grex

T_U = uncorrected tenacity* in g per grex

E = elongation at break in percent

Relative knot tenacity and extensibility. These values are expressed as percentages of original uncorrected tenacity and elongation at break respectively.

Fiber length and fineness after mechanical conditioning. The length increase in percent is shown immediately after mechanical conditioning and after one hour relaxation. The difference between these values represents shrinkage during relaxation. The fineness after mechanical conditioning and relaxation is shown as new fineness.

* Uncorrected tenacities based on initial fineness are plotted in all the figures except for the four curves of the wool yarn demonstrated in Figure 7B.

TABLE I
CHARACTERISTICS AND STRESS-STRAIN PROPERTIES OF FIBERS TESTED

	Wool Single Fiber	Worsted Wool Yarn 28.4/1	Mohair Single Fiber	Mohair Yarn 28/1	Human Hair Single Fiber	Casein Multi- fila- ment 300/40	Vicara Single Fiber	Silk Multi- fila- ment
CHARACTERISTICS								
1. Original fineness in grex	15.5	315	13.4	312	50.2	333	3.33	117
2. Staple length in inches	3.75	2.85	4.38	4.41				
3. Twist, turns per inch		20.9		16.0		4.5		4.1
4. Density	1.34	1.34	1.34	1.34	1.34	1.29	1.24	1.38
ORIGINAL BEHAVIOR								
5. Tenacity at break								
a) Uncorrected in g/gx	1.10*	0.84	1.39*	0.48	1.82	0.78	0.86	4.02
Coefficient of variation in %	28.3*	15.5	53.0*	20.7	3.7	4.3	6.0	2.5
b) Corrected in g/gx	1.44*	1.12	1.88*	0.56	2.63	1.10	1.14	4.89
6. Elongation at break in %	31.0* ⁵⁾	33.2	35.0*	17.8	44.4	41.0	32.3	21.8
Coefficient of variation in %	13.9*	17.2	15.0*	29.7	8.2	7.8	4.6	5.4
KNOTTED								
7. Tenacity at break								
a) Uncorrected in g/gx		0.75		0.52	0.98	0.76	0.79	3.30
Coefficient of variation in %		19.4		28.3	11.3	3.3	11.5	8.9
b) Relative knot tenacity in %		90		108	54	98	92	82
c) Corrected in g/gx		0.86		0.62	1.15	1.06	0.91	3.71
8. a) Elongation at break in %		27.3		19.9	13.1	39.0	28.2	12.7
Coefficient of variation in %		31.3		25.8	35.2	7.2	14.1	16.1
b) Relative knot extensibility in %		82		111	30	96	87	58
MECHANICALLY CONDITIONED								
9. Increased length in % ¹⁾								
a) Immediately		16.9			23.8	25.1	20.0	11.9
b) After relaxation		10.6			21.2	23.8	17.5	11.2
10. Shrinkage in % during relaxation ¹⁾		6.3			2.6	1.3	2.5	0.7
11. New fineness in grex		285			41.4	269	2.84	106
12. Tenacity at break								
a) Uncorrected in g/gx ²⁾		0.82			1.79	0.82	1.02	3.98
Coefficient of variation in %		8.7			5.3	3.9	13.3	3.4
b) Uncorrected in g/gx ³⁾		0.91			2.17	1.02	1.20	4.40
c) Corrected in g/gx		1.14			2.58	1.16	1.32	4.71
13. Elongation at break in % ⁴⁾		25.3			18.9	13.7	10.1	7.0
Coefficient of variation in %		17.5			6.8	11.5	16.3	11.8
WET								
14. Tenacity at break								
a) Uncorrected in g/gx	0.66*	0.48	1.14*		1.34*	0.28	0.61*	3.21
Coefficient of variation in %	48.2*	13.1	29.4*		15.6*	7.3	21.0*	3.3
b) Relative wet tenacity in %	60*	58	83*		78*	36	68*	80
c) Corrected in g/gx	0.96*	0.72	1.80*		2.02*	0.41	0.84*	4.27
15. a) Elongation at break in %	45.0* ⁵⁾	49.0	57.5*		51.3*	44.2	38.8*	33.1
Coefficient of variation in %	15.8*	6.5	12.7*		12.6*	6.5	20.0*	4.4
b) Relative wet extensibility in %	145*	148	165*		116*	112	112*	151

REMARKS: 1) Correlated to the original length
2) Correlated to original fineness before mechanical conditioning (listed in line 1)
3) Correlated to new fineness after mechanical conditioning (listed in line 11)
4) Correlated to the new length after mechanical conditioning
5) Without crimp
* Values obtained using the Sookne-Harris tester

Relative wet tenacity and extensibility. The ultimate values of wet fibers are expressed as percentages of the corresponding original values.

1. Tests of Original Fibers

The following facts are apparent from the results demonstrated in Figure 4 and Table I:

a. The stress-strain relationship, tenacity, and elongation at break of silk are very different from those of the other protein fibers as would be expected from the marked chemical and structural differences which exist between them.

b. The human hair sample is the strongest and the most extensible of the keratin fibers tested.

c. The initial flat portion on the stress-strain curve of the wool single fiber and the slow increase of stress at the beginning of the stretching process reflects the crimp (approximately 4 percent). Such a flat portion is not apparent on the stress-strain curve of the wool yarn.

d. The initial modulus, the yield point, and the tenacity at break for wool and mohair yarns are lower than the corresponding values for the single fibers, showing the influence of yarn structure. The low extensibility of the mohair yarn indicates slippage of single fibers as a result of the smooth surface, absence of crimp, and relatively low twist.

e. A decrease of tenacity with increasing elongation, after the yield point is passed, is characteristic for the man-made fibers, casein and Vicara. This decrease is due to "plastic flow" which is not present to such an extent in natural protein fibers.

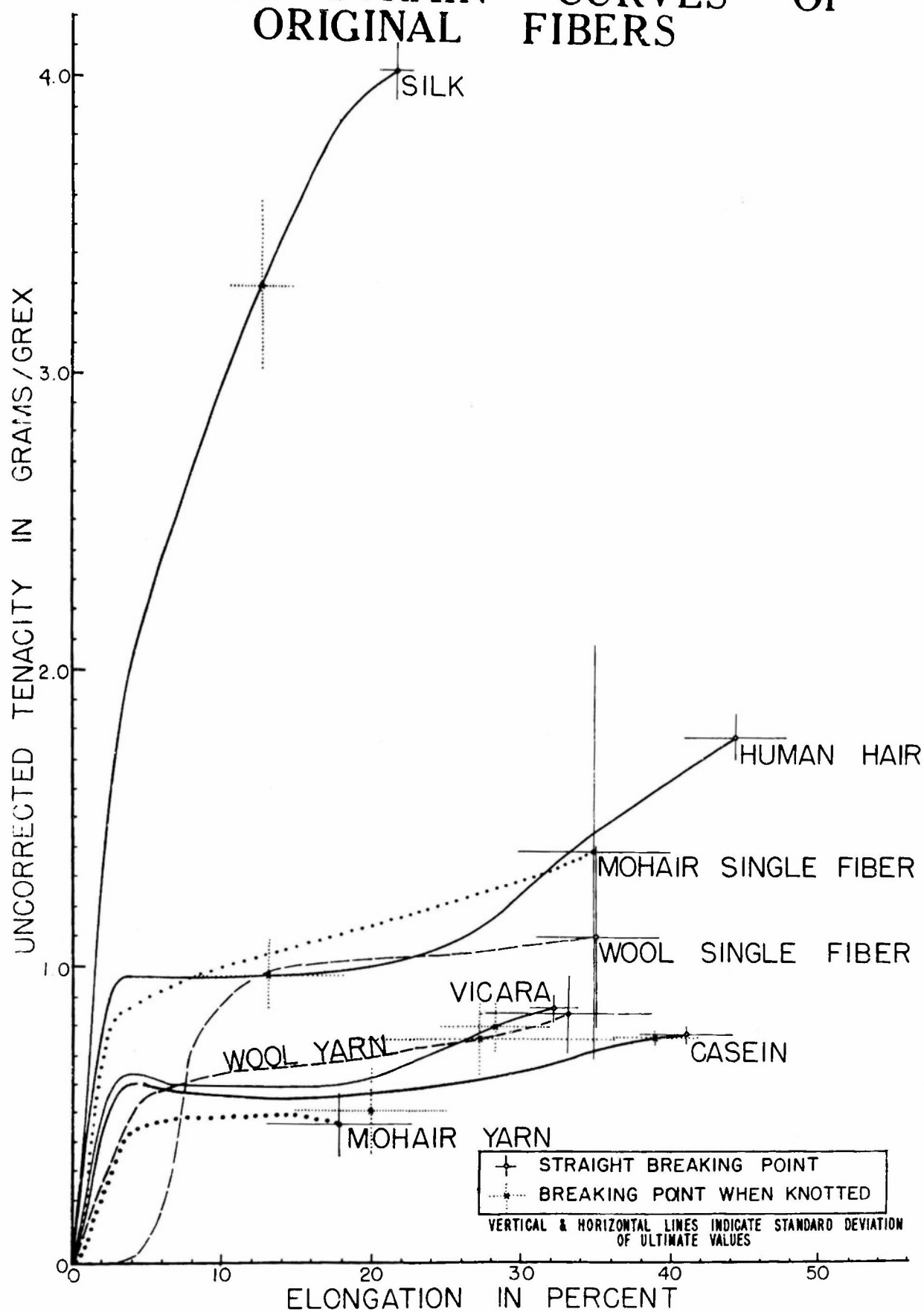
f. Except for mohair single fibers, the uniformity of the fibers tested is reasonably good when based upon the variation of ultimate stress and strain values.

2. Knot Tests

Most of the specimens tested when knotted fail near the original breaking point as shown on the stress-strain curves (Figure 4), and by the relative knot tenacities and extensibilities (lines 7b and 8b, Table I). (Knot data are also demonstrated by a dotted horizontal line in the rectangular and quadratic graphs of Figures 8 and 9 which will be described later.)

Wool yarn, silk and casein multifilament, and Vicara single fiber have high relative knot tenacities, namely between 82 and 98

FIG. 4
STRESS - STRAIN CURVES OF
ORIGINAL FIBERS



percent. If both decrease of tenacity and extensibility are considered, only silk and human hair show appreciable losses when knotted. The low relative knot values of human hair (54 and 30 percent, respectively) indicate a notable weakening of this otherwise strong fiber when bending and shearing forces are present. The surprisingly high values of mohair yarn (108 and 111 percent, respectively) are a result of restricted slippage by the knot. The staple fibers are long, having an average length of 4.38 in. and a premature slippage is prevented by the presence of a knot.

It can be seen from Figure 4 and from the data in Table I that some ultimate values for knotted fibers do not show significantly higher variance than corresponding values for unknotted fibers.

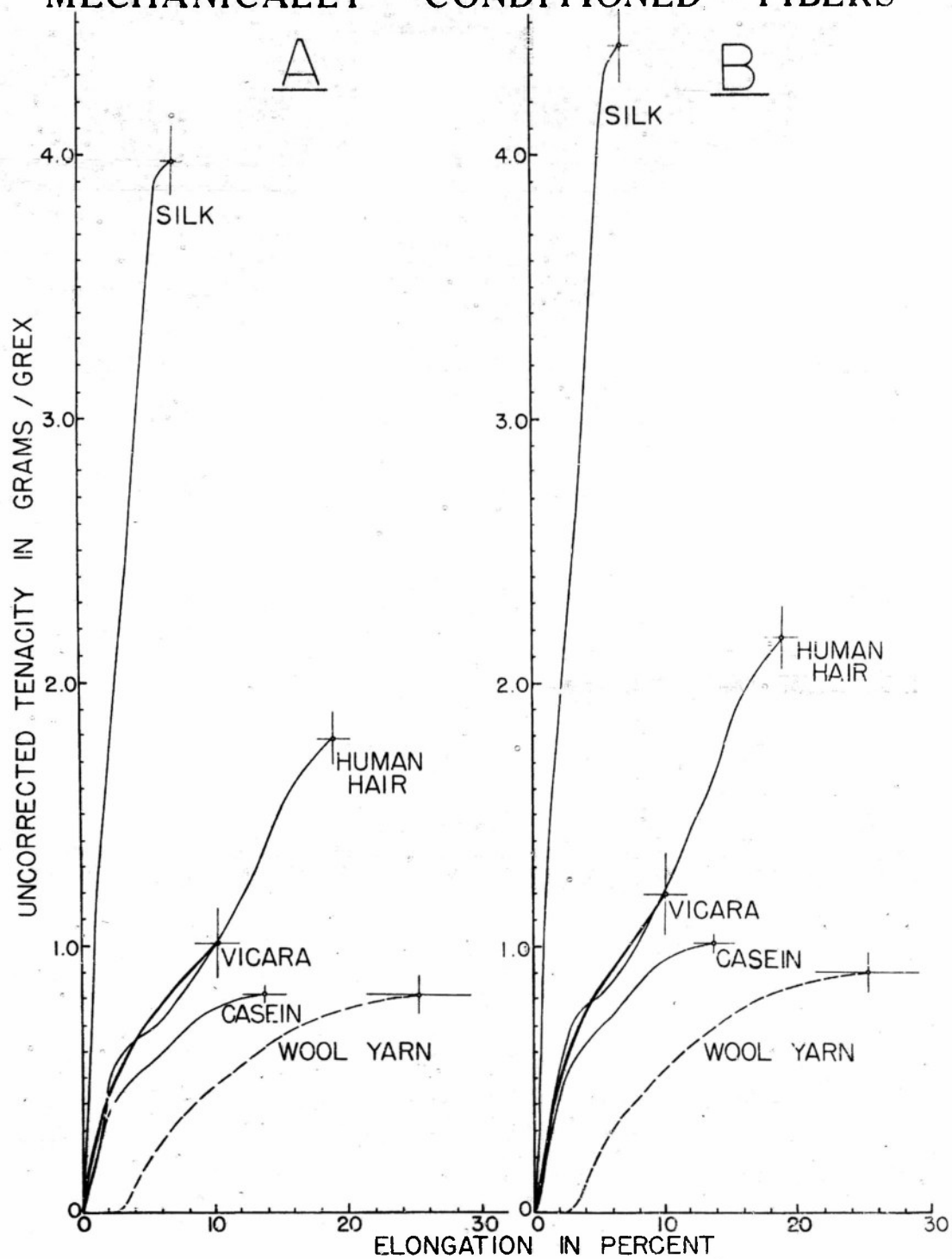
3. Tests of Mechanically Conditioned Fibers

The following five materials were tested after mechanical conditioning: single fibers of human hair and Vicara, multifilaments of silk and casein, and the worsted wool yarn.

A marked increase of the original fiber length and consequently a decrease in the fiber grex was observed in every case after repeated stresses. The stress-strain relationship of mechanically conditioned fibers is demonstrated in two ways: in Figure 5A the uncorrected tenacities are correlated to the original fineness before mechanical conditioning, and in Figure 5B they are correlated to the new fineness after mechanical conditioning; in each case the uncorrected tenacities are plotted against elongation expressed as percentage of the new length after mechanical conditioning.

The definite yield point disappeared from the stress-strain curves of wool and Vicara, and was barely perceptible for human hair and casein, after repeated stresses. The shape of the stress-strain curves of mechanically conditioned wool, human hair, casein and Vicara fibers thus approaches that of silk in its original state where no pronounced yield point is observable. Dispersion of ultimate values for mechanically conditioned samples is in most cases approximately the same as for the original fibers.

FIG. 5
STRESS - STRAIN CURVES OF
MECHANICALLY CONDITIONED FIBERS



A. TENACITY VALUES BASED ON ORIGINAL FINENESS.
 B. TENACITY VALUES BASED ON NEW FINENESS (AFTER MECHANICAL CONDITIONING)
 VERTICAL & HORIZONTAL LINES INDICATE STANDARD DEVIATION OF ULTIMATE VALUES.

The uncorrected tenacities of mechanically conditioned fibers correlated to the new fiber fineness (line 12b, Table I) are always higher than the original tenacities (line 5a). Except for Vicara, practically identical values appear, however, when uncorrected tenacities of mechanically conditioned fibers correlated to the original fiber fineness (line 12a) or corrected tenacities (line 12c) are compared with the corresponding original tenacities (lines 5a and 5b). They show that no loss in tenacity occurred by mechanical conditioning as a result of fatigue, despite the severity of the conditions under which the repeated extensions were performed. The higher tenacity of Vicara indicates a stress-hardening (approximately 17 percent). It is the result of alignment of long-chain molecules in the amorphous and crystalline portion of this fiber. In contrast to this behavior the new extensibility of all the five fibers tested decreased markedly after mechanical conditioning (compare lines 6 and 13). This phenomenon was caused by the elimination of permanent set as will be shown later in the investigation of recovery behavior.

One may be surprised at the initial flat portion on the stress-strain curve of the wool yarn after repeated stresses, corresponding to an elongation of approximately 2.8 percent. Since the individual fibers were aligned in the stretching procedure, their crimp, originally present in the yarn structure, was eliminated by mechanical conditioning. The ability of wool fibers to curl again after release of the tension was retained, however. They recurled during the relaxation time of one hour, re-forming loose connections with neighboring fibers. These new connections were broken easily and the recovered crimp was removed in the succeeding tensile test at a low, almost undetectable tension, causing the initial flat portion on the stress-strain curve of the mechanically conditioned yarn.

This recurling and mobility of the individual wool fibers in the yarn structure is also revealed by the considerable shrinkage of the wool yarn after cycling in comparison to that of human hair. The recovery behavior of both wool yarn and human hair fiber is similar. Their length increased markedly after mechanical conditioning i.e., by 16.9 and 23.8 percent of their original length (line 9a).

These length increases were, of course, higher than calculated from the permanent set values in a single loading* due to the fact that a larger portion of the elongation became "nonrecoverable" in 50 cycles than in a single loading step.

During the relaxation period the wool yarn shrank 6.3 percent of its initial length, and expanded laterally, and the hair shrank only 2.6 percent (line 10).** The effect of repeated stresses on the wool yarn is thus to a considerable extent irreversible upon relaxation because of the capacity of the individual wool fibers to contract by delayed recovery and to recurl. For the straight hair fibers on the other hand, delayed recovery is the only source of longitudinal contraction during relaxation. Despite this shrinkage, the length of both fibers remained greater than originally, their length increase being 10.6 and 21.2 percent respectively (line 9b). Thus the final length of the wool yarn was practically identical to that expected from its elongation after a single loading, indicating the complete return of the individual wool fibers to their position before cycling. In contrast to this, the hair after relaxation still remained much longer than calculated from a single loading step (21.2 percent longer compared to 13.5 percent).

* The permanent set of both fibers (as shown in the upper quadratic graphs of Figure 9, page 31) is approximately 38 percent of their actual elongation at 80 percent of the elongation at break, to which level the fibers were repeatedly extended (i.e., to actual elongations of 26.6 and 35.5 percent respectively). From these values length increases of 10.1 percent for the wool yarn and 13.5 percent for hair can be calculated after a single loading step.

** The difference between the two shrinkages, 3.7 percent, is similar to the elongation value (2.8 percent) of the initial flat portion reflecting the rebuilt crimp on the stress-strain curve of the mechanically conditioned yarn.

The behavior of wool yarn after mechanical conditioning is analogous to the "aging effect" of wool recently described by von Bergen and Wakelin. [32] In a systematic investigation of alterations during worsted processing they observed that single fibers (taken out of roving and yarn) increased after resting. They found also that the "maximum load" of the stress-strain curves for rovings was higher after resting. These changes took place rapidly at first until fairly constant values were reached approximately in three weeks. Moist heat, steaming at 185 F, accelerated the "aging" and it was interpreted as a relaxation phenomenon due to the "gradual return of crimp."

The alterations of protein fibers due to mechanical conditioning reveal the changed morphology (fiber rearrangement, elimination of crimp) as well as the changed fine structure (orientation of crystallites, slippage of long-chain molecules, and formation of new cross-bonds). They are, of course, most pronounced immediately after the cycling procedure and they diminish during the relaxation period indicating that morphological and fine structural changes attained by mechanical conditioning are partly reversible. [28] The relaxation time of one hour as practiced in this study was considered to be sufficient for an approximation of ultimate values. It was probably not long enough to attain equilibrium for the wool yarn in view of the delayed recovery and mobility of the individual wool fibers, both known to be high. Additional progression of relaxation phenomena could therefore take place in the wool yarn in subsequent hours or days after mechanical conditioning.

4. Wet Tests

Yarns of wool casein and silk were tested wet with the Instron tensile tester, while the Sookne-Harris tester served for wet tests on single fibers of wool, mohair, human hair and Vicara. As shown in Figure 6, swelling in water has the effect of increasing the extensibility of the fibers

and of weakening them. This is evidenced by lower breaking tenacities, lower yield points, and lower initial moduli. Mohair, silk and human hair, having relative wet tenacities between 78 and 83 percent, are less affected by water than wool, casein, and Vicara whose wet tenacities are between 36 and 68 percent. This behavior is not at all surprising for silk, in view of its high crystallinity and high degree of orientation. Both decrease the swelling of fibers which occurs mostly in the amorphous part of high polymers. The resistance of the human hair and mohair specimens to swelling is due partly to their morphology and chemical composition. The ratio between the resistant cuticle cells and the easily affected cortical cells is higher for hair than for wool. Besides this, hair keratin has a higher cystine content (19.0 percent) than wool keratin (12.2 percent) and the sulfur cross-bonds of cystine increase the stability of protein molecules. Zahn [38] demonstrated that human hair has a higher "softening point" than wool - 203 F compared to 183 F when measured by 20 percent "supercontraction" (shrinkage) in a 1:1 phenol-water mixture. The action of heat is in many cases similar to that of swelling agents and the higher stability of hair to heat corresponds to its greater resistance to the swelling action of water. The stress-strain curves of the wool yarn and of the wool single fiber are similar when wet, except for their initial parts. The flat portion on the curve for the wet single fiber again indicates the crimp (approximately 4.9 percent).

B. Tensile Recovery

1. Recovery of Wool Yarn

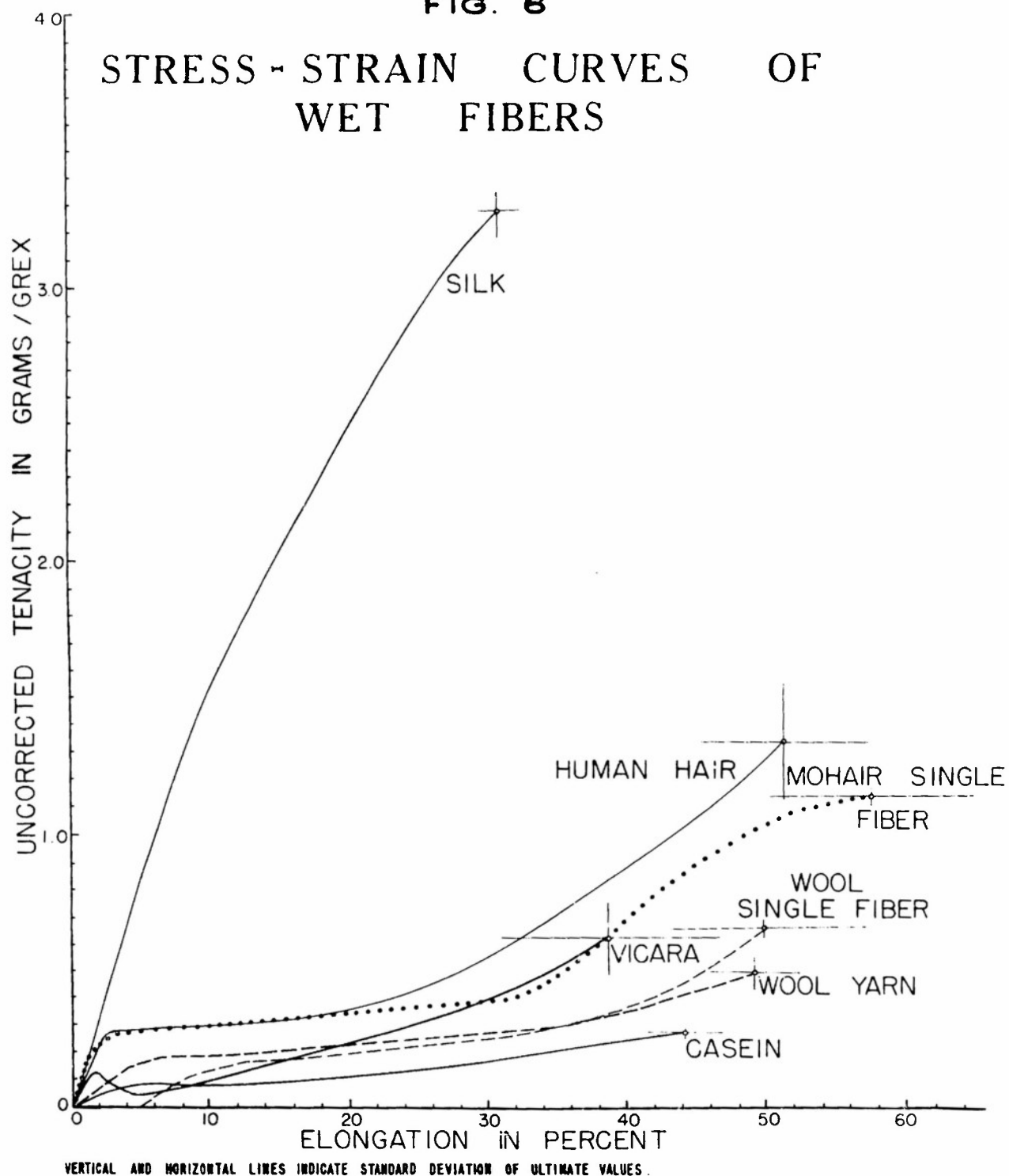
The elongation components of the original wool yarn are shown by four curves in Figure 7A in which their actual values are plotted against uncorrected tenacities based on initial fineness.

The following observations can be made from these four curves:

a. Below the yield point the elongation is completely recoverable, the immediate recovery being slightly greater than the delayed recovery. Actual values of immediate elastic recovery increase steadily up to the breaking point.

FIG. 6

STRESS - STRAIN CURVES OF WET FIBERS



b. A considerable increase of delayed recovery takes place at the yield point. At the breaking point it is much higher than immediate recovery, even slightly exceeding the permanent set.

c. Permanent set starts at the yield point, increasing continuously and rapidly up to the breaking point.

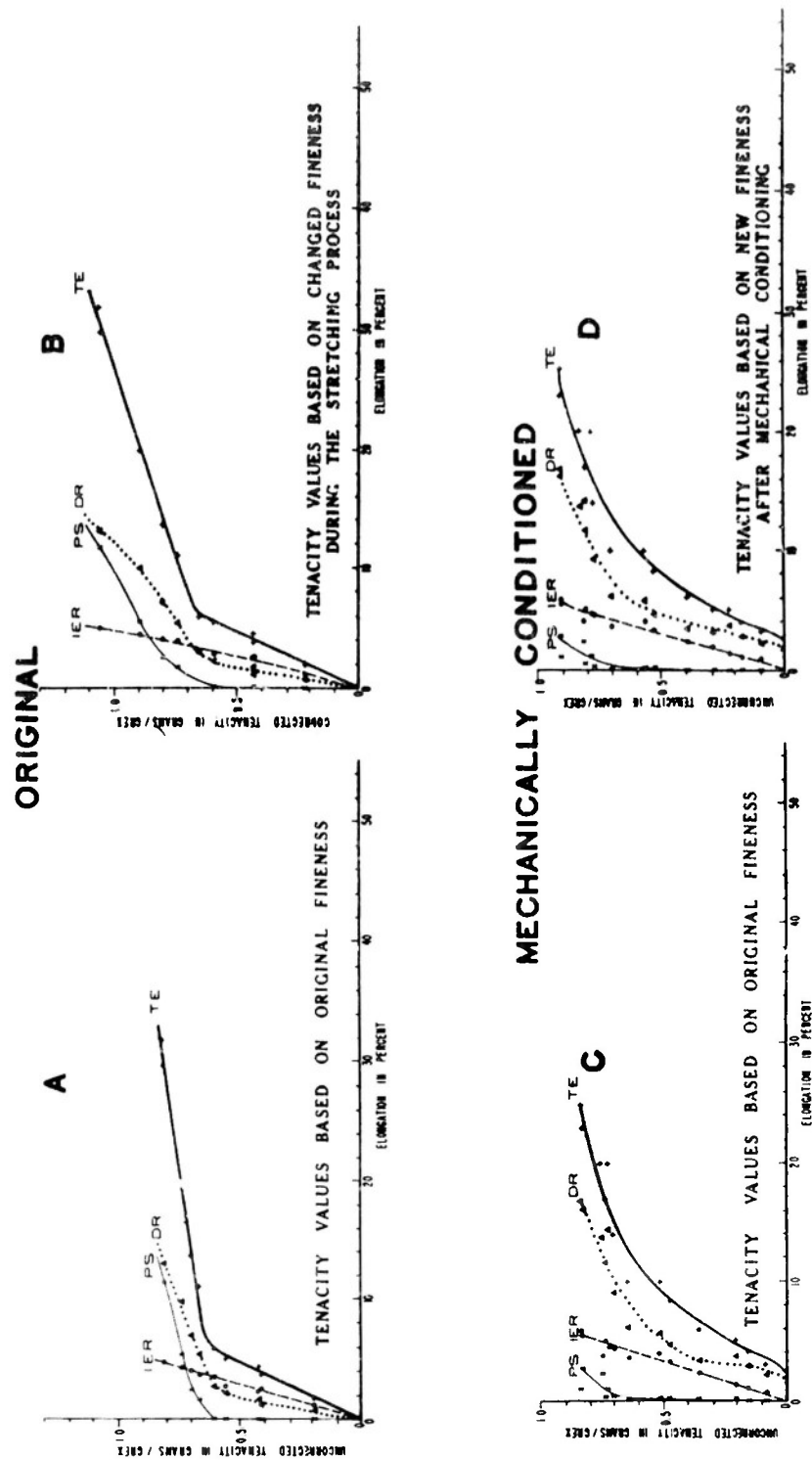
The recovery of the wool yarn is thus characterized by a relatively high delayed recovery similar to that of drawn nylon and Vinyon CF-HST, but not observed to this extent in other textile fibers. [29] Most fibers (casein, acetate, silk, viscose) show predominance of permanent set near the breaking point, while others (Fiberglas, Fortisan) have high recovery but low extensibility near the point of rupture.

The actual values of the three strain components demonstrated in Figure 7A are plotted in Figure 7B against corrected tenacities based on the changed fineness during the stretching process. Except for an increase in tenacity and slight differences in the initial modulus, the results using corrected tenacities (Figure 7B) are very similar to those using uncorrected tenacities (Figure 7A). Although a presentation by corrected tenacities has some advantages it is rarely used in testing textile fibers. [28]

Since the tensile recovery of fibers is altered significantly after mechanical conditioning and when wet, the recovery of the wool yarn was also investigated under these conditions. The curves in Figure 7C and D show the actual values of the three elongation components after repeated stresses. In both cases uncorrected tenacities are plotted against identical elongation values. In Figure 7C the tenacity is based on the original fineness (315 grex) and in Figure 7D on the new fineness after mechanical conditioning (285 grex). It again appears from both graphs that the permanent set starts at the yield point which is, in this case, fairly close to the breaking point. The permanent set at the breaking point is low compared to that of the original fiber (3.0 percent versus 13.6 percent). Even this low permanent set is due to the fact that the specimens had been repeatedly extended to only 80 percent of the elongation at break; therefore, permanent set near the breaking point was not eliminated. Delayed recovery is the predominant elongation component of mechanically conditioned wool yarn at the breaking point. It can be seen that the curves representing immediate and delayed recovery are almost identical to those of the original yarn except for the initial flat section on the curve for delayed

ELONGATION COMPONENTS OF WOOL STAPLE YARN

FIGURE 7



recovery after repeated stresses which also appears on the stress-strain curve (the heavy curve in Figure 7C and D). If the initial flat portion (2.8 percent elongation) is subtracted from the ultimate value of delayed recovery (16.7 percent), the remaining value (13.9 percent) is close to the delayed recovery of the original sample (14.6 percent).

It is remarkable that the elongation at low strain values (shown by the flat portion) does not recover immediately, as it does for most fibers but requires some relaxation time.* The relatively high delayed recovery at small, almost undetectable tensions indicates the creeping return of the aligned individual fibers to their former crimped state and it demonstrates again the mobility of wool fibers in the yarn structure.

The elongation components of the wet wool yarn using a wet fabric cover were shown in Figure 3A. Unlike the behavior of dry wool, only a very small permanent set appears at high stress and strain values (2.4 percent at the breaking point). Elongation of wet wool is thus almost entirely recoverable, confirming the observations of Speckman, [27] Harris and Brown, [9] Sookne, [26] Maillard et al., [11] and of other investigators. This behavior is characteristic for natural keratin fibers and not duplicated to such extent by any other textile materials. It is remarkable that the permanent set of wet wool does not start at the very low yield point (at a tenacity of 0.17 g per grex and an elongation of 5.7 percent), but at a much higher point (approximately at 0.40 g per grex tenacity and 40 percent elongation) known as the elastic limit.

* The interpretation of the initial flat portion as delayed recovery may appear questionable from the cycling tests performed since an initial flat portion is also observable on the second loading curve of mechanically conditioned yarns. Tension is indicated here only after an elongation in excess of approximately 3 percent. The length of this flat portion in the second loading cycle, however, is identical to that of the first cycle. Besides this, a more or less pronounced slack in the mechanically conditioned yarn is observable in performing cycling tests for small elongations immediately after removal of the tension. The slack disappears progressively during the relaxation time of five minutes and this manifests the creeping recovery. At elongations higher than 15 percent a considerable slack remains in the mechanically conditioned yarn after the relaxation time and the length of the flat portion in the second loading curve is now increased. This length corresponds to permanent set.

Differentiation between the yield point and elastic limit is therefore necessary, although the two terms coincide in most cases.* Figure 3A shows that both the immediate and delayed recovery of wet wool increase substantially upon passing the yield point and that the overwhelming portion of the total elongation at the breaking point consists of delayed recovery, having a value of 39.2 percent, as compared to 14.6 percent for dry wool.

Recovery behavior of the wool yarn shown in Figure 7A can be demonstrated more conveniently by two rectangular or quadratic graphs as described previously. As shown in Figures 8 and 9, these graphs reveal the following facts:

a. The wool yarn is highly recoverable in its original state, since areas signifying recovery (immediate elastic recovery as well as delayed recovery) occupy the major portions of the graphs.

b. Immediate elastic recovery exceeds delayed recovery at low strain and stress values. Its relative value decreases rapidly when the elastic limit is passed.

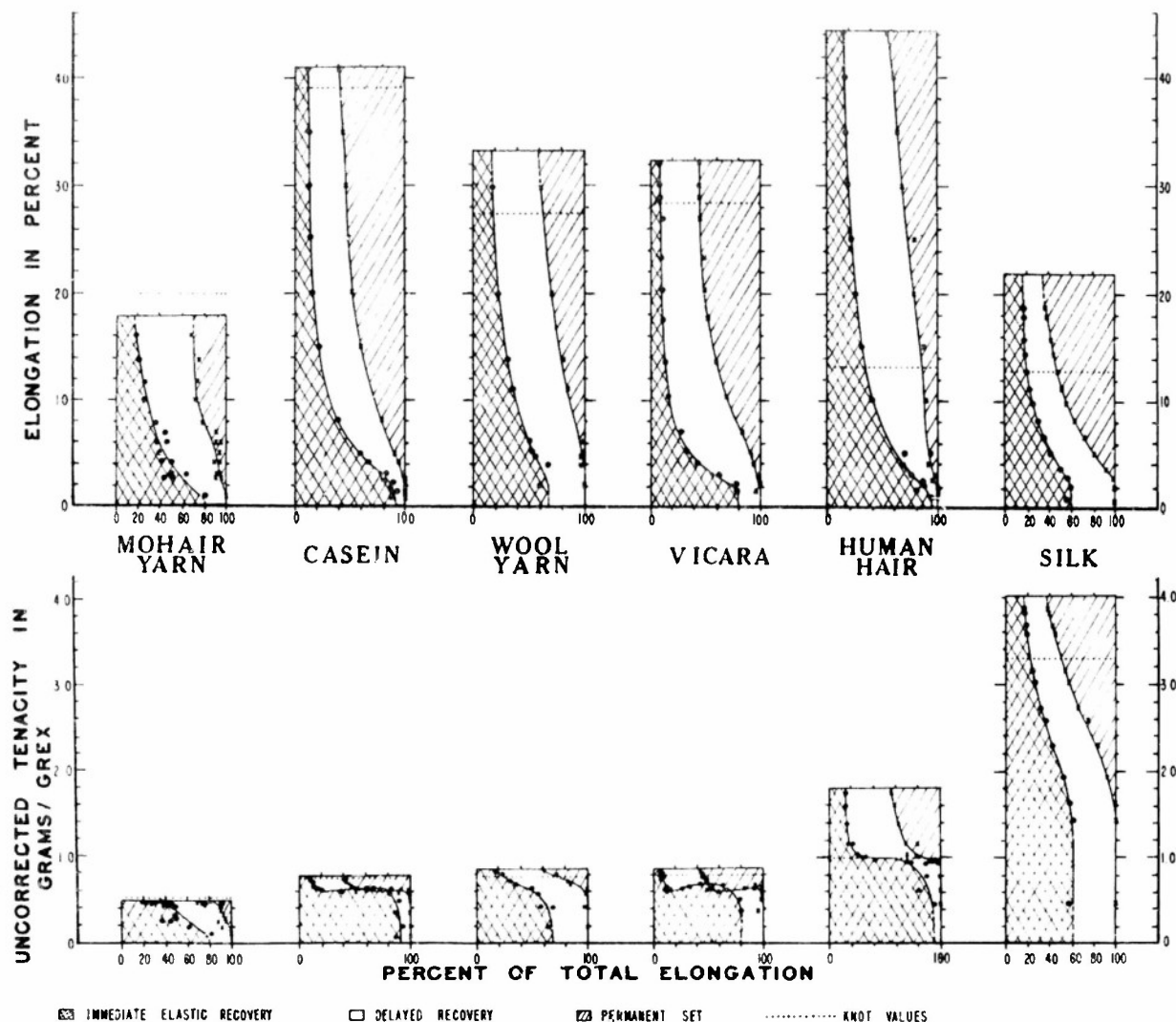
c. Only a small percentage (15 percent) of the total elongation does recover immediately near the breaking point; a larger percentage (44 percent) is primary creep which exceeds even the permanent set (41 percent). The relative values of delayed recovery do not change substantially during the stretching process.

A convenient comparison of the recovery behavior under very different test conditions is possible from the quadratic graphs since the considerable differences in ultimate values have been eliminated intentionally. These graphs (Figure 9) show clearly that even the relatively small permanent set of the original wool yarn disappears almost entirely as a result of repeated stresses or of wetting. Recovery is, however, immediate only to a small extent in both cases. Values of immediate elastic recovery do not change substantially during the stretching process after repeated stresses, showing a slight maximum at approximately 30 percent of the elongation at break and at 60 percent of the breaking tenacity. They decrease slightly, however, at progressively increasing stress and strain beyond the yield point when the yarn is wet.

* At the yield point a more or less pronounced change in the relationship between stress and strain occurs. Elastic limit designates that point of the stress-strain curve where non-recoverable elongation (permanent set) starts.

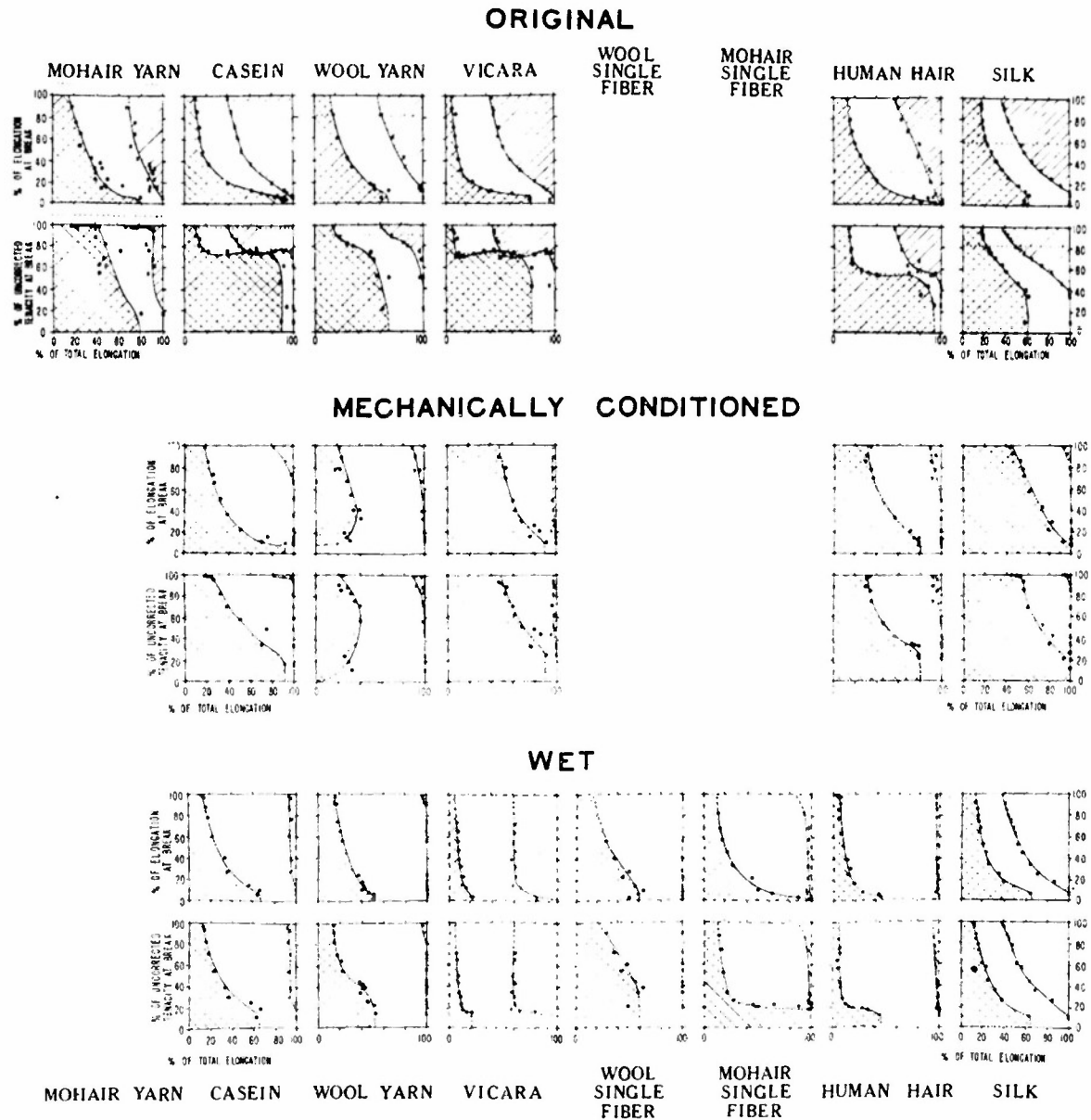
FIG. 8

RECOVERY BEHAVIOR OF ORIGINAL FIBERS

2. Recovery of Other Protein Fibers

Recovery of protein fibers in their original state may be discussed first by considering the rectangles of Figure 8, arranged (as indicated by the height of the graphs in the lower set) in order of increasing uncorrected tenacity at break. The recovery of natural keratin fibers (wool, mohair, and human hair) appear similar in the rectangles of the upper row despite marked differences in height representing their extensibility. Nearly identical values of the three elongation components are apparent at the same level of elongation for these fibers. These fibers are characterized by relatively small permanent set and high recovery even at the breaking point, the major part of the recovery being delayed. The recovery of Casein and Vicara differs slightly from these fibers in that the nonrecoverable portion of the elongation is increased, recovery being lower at the breaking point.

FIG. 9
RECOVERY BEHAVIOR OF FIBERS TESTED



LEGEND

IMMEDIATE ELASTIC RECOVERY	DELAYED RECOVERY	PERMANENT SET
KNOT VALUES		
SOLID FRAMES INSTRON TENSILE TESTER		DASHED FRAMES SOOKNE-HARRIS TESTER

The recovery of natural keratin fibers is very different, however, from that of silk which shows a higher portion of permanent set (especially near the breaking point) and also significantly diminished delayed recovery. The immediate elastic recovery of silk is nearly equal to that of the other fibers tested when compared at the same elongation level.

The recovery behavior of the fibers tested originally, after mechanical conditioning, and when wet is also shown by three sets of quadratic graphs in Figure 9, which are arranged in the order of increasing tenacity in the original state. The tenacities are based upon the "new" fineness of the mechanically conditioned samples and on the original (dry) fineness of the wet samples.

Numerical data of actual and relative values of the three elongation components at the breaking point are also presented in Table II in the original state, after repeated stresses, and when wet. The results of recovery tests using the Sookne-Harris tester are presented by quadrats with a dashed frame to differentiate them from the more representative tests made on the Instron.

The quadrats representing mechanically conditioned fibers demonstrate that permanent set is almost entirely eliminated from the five fibers tested after repeated stresses. This behavior is similar to that of other textile fibers [28] and it is a result of their markedly diminished extensibility after mechanical conditioning. There are differences, however, in the recoveries, with that of Vicara and silk being to a great extent immediate, and that of casein, wool, and human hair being predominantly delayed, especially near the breaking point.

The quadrats representing wet fibers show the following facts:

a. Samples of casein, wool, mohair and human hair have similar recovery characteristics when wet in that permanent set disappears almost entirely and delayed recovery is predominant.

b. The small permanent set observable on wet wool yarn near the breaking point does not appear on wet wool single fibers despite the limitations of the Sookne-Harris tester which necessitated a lower jaw speed to be used in these tests. The permanent set of wet wool yarn apparently has its origin in fiber slippage at high tensions.

c. The recovery of wet Vicara is interesting since the elongation components have approximately the same relative values from almost the beginning of stresses and strains up to the breaking point. Only a small percentage of the elongation is immediately recoverable while the predominant portion recovers through "creep." A relatively high

TABLE II
RECOVERY BEHAVIOR OF FIBERS TESTED

	Wool Single Fiber ¹⁾	Wool Yarn	Mohair Single Fiber	Mohair Yarn	Human Hair Single Fiber	Casein Multi- fila- ment	Vicara Single Fiber	Silk Multi- fila- ment
<u>Actual Values of Elongation Components in Per Cent</u>								
<u>Original</u>								
Immediate elastic recovery		5.0		3.0	6.2	4.1	2.3	3.4
Delayed recovery		14.6		9.5	18.6	11.9	11.6	4.5
Permanent set		13.6		5.3	19.6	25.0	18.4	13.9
<u>Mechanically Conditioned²⁾</u>								
Immediate elastic recovery		5.6			6.0	2.6	4.8	3.2
Delayed recovery		16.7			11.2	8.4	5.0	3.3
Permanent set		3.0			1.7	2.7	0.3	0.5
<u>Wet</u>								
Immediate elastic recovery	7.2*	7.4	7.0*		3.6*	6.2	1.9*	4.0
Delayed recovery	37.8*	39.2	42.0*		45.6*	34.9	21.0*	8.4
Permanent set	0*	2.4	8.5*		2.1*	3.1	15.9*	20.7
<u>Relative Values of Elongation Components in Per Cent</u>								
<u>Original</u>								
Immediate elastic recovery		15		17	14	10	7	16
Delayed recovery		44		53	42	29	36	20
Permanent set		41		30	44	61	57	64
<u>Mechanically Conditioned²⁾</u>								
Immediate elastic recovery		22			32	18	48	46
Delayed recovery		66			59	62	49	47
Permanent set		12			9	20	3	7
<u>Wet</u>								
Immediate elastic recovery	16*	15	12*		7*	14	5*	12
Delayed recovery	84*	80	73*		89*	79	54*	25
Permanent set	0*	5	15*		4*	7	41*	63

REMARKS: 1) Without crimp
2) Correlated to the new length after mechanical conditioning
* Values obtained using the Sookne-Harris Tester

percentage of the elongation is non-recoverable, however, which is in contrast to the behavior of wet casein and wet keratin fibers.

d. The areas of the elongation components in the quadrats for silk in its original state and when wet are not very different from each other despite marked differences in the stress-strain relationship and in the ultimate values. (Figures 4 and 6). Different actual but similar relative values

of elongation components at the breaking point of silk in its original state and when wet are also apparent from the numerical data shown in Table II. Relative values of recovery remain thus unchanged by wetting despite the marked weakening of silk by swelling.

An increase in recovery is characteristic for most protein fibers when swollen. This is very pronounced for natural keratins and it can be also observed in the regenerated protein fibers. Silk, however, does not have this property, remaining relatively unchanged.

VII SUMMARY

Knowledge of the tensile behavior of the protein fibers - wool, mohair, hair, casein, vicara and silk - has been extended to include quantitative data on their elastic recovery. Needed information has also been obtained by investigating the influence of repeated stresses and of swelling. It has been shown that the recovery of wool in its original state is exceptional, that after mechanical conditioning it is similar to that of other fibers, and that it is unique when wet. The investigation of a mechanically conditioned wool yarn revealed that although the crimp of the individual wool fibers is temporarily removed by stretching, it is rebuilt spontaneously during the relaxation period. The restoration of crimp manifests the ability of individual wool fibers to recur and to change their position with relation to each other within the yarn structure after the strain or stress has been released. This mobility of wool fibers, the high elastic recovery and the improvement in recovery when wet are properties not present (at least to such an extent) in other textile fibers. These characteristics are basically responsible for the resilience, crease retention, wrinkle resistance, excellent dimensional stability, hand and liveliness of woolen fabrics.

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